

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Applicant(s): John W. Locher, et al.

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Title: SINGLE CRYSTALS AND METHODS FOR FABRICATING SAME

App. No.: 10/820,468 Filed: April 8, 2004

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## DECLARATION UNDER 37 C.F.R. §1.132

Sir, I hereby declare and state:

1. I am a joint inventor of the subject matter presently claimed in the above-identified patent application.
2. I received my undergraduate degree in Chemistry from University of North Carolina in 1976 and received my masters degree in Mechanical Engineering and Materials Science from Duke University in 1979.
3. Since 1979, I have been involved in the research and development of crystal growth, particularly, single crystal materials including melt-based processing of single crystal sapphire. Since 1987, I have been employed by Saint-Gobain Ceramics & Plastics, Inc. (or its predecessor companies), during which time I have primarily been engaged in research and development of crystal growth technologies.
4. I have reviewed the substance of the Office Action mailed March 9, 2006, as well as the cited prior art reference JP 57095899 A to Takahashi et al.

5. Background. The claimed invention is directed to a large-sized sapphire single crystal, comprising a single crystal sheet having a width not less than 15 cm, and a length greater than the width. Independent claim 1 further recites that the thickness is not less than 0.5 cm, independent claim 6 further recites that the sheet has a variation of thickness of not greater than 0.2 cm, and independent claim 11 combines the features of claims 1 and 6. Independent claim 12 recites an as-grown single crystal sheet, including a main body portion and a neck portion, the neck portion having a uniform construction as quantified by a max  $\Delta_t$  of 4.0cm.

The claimed invention provides for large-sized sapphire single crystals in the form of single crystal sheets, and represents a notable advancement over the state of the art. In this respect, the attention of the PTO is drawn to the previously submitted technical article entitled "Large Diameter Sapphire Window From Single Crystal Sheets" from the Proceedings of the 5<sup>th</sup> DOD Electromagnetic Window Symposium. The article describes an attempt to scale-up EFG-grown sapphire sheets to dimensions of 30.5 cm wide X 48 cm long X 0.25 cm thick. Although the article describes formation of such a sized single crystal sapphire sheet, the technology described therein is limited, particularly confined to formation of what is essentially medium-sized sapphire single crystal sheets, by virtue of the limited thickness achievable by the described technology. Indeed, the claimed invention has a thickness at least twice that disclosed by the article, having an attendant mass twice that of the article (for the same width and length).

In an effort to provide next-generation sapphire single crystal sheets, my co-inventors and I have developed a process flow incorporating notable features, such as use of a high aspect ratio crucible (see FIG. 4 and paragraph 28 of the present specification), incorporation and manipulation of a gradient trim system for dynamically adjusting the thermal gradient along the length of the die through which the sheet is formed (see FIGs. 1-2, gradient trim system 50, paragraphs 25 and 36, for example), incorporation of a particular heat shield assembly providing a baseline thermal profile (see shield assembly 26, FIGs. 1-3 and paragraphs 21 and 26), as well as other structural and process-oriented features. Stated alternatively, we have developed enabling technology to provide next-generation large-sized single crystal sapphire sheets by EFG, having a high level of uniformity.

6. High Aspect Ratio Crucible. Use of a high-aspect ratio crucible is of particular consequence in connection with the ability to form large-sized sapphire single crystal sheets having low thickness variation as well as uniform construction quantified by  $\Delta_T$ . In the development of the claimed invention, we shifted from use of a conventional crucible that has a generally circular or round contour, to a crucible having a high aspect ratio, such as an aspect ratio of 2:1 or greater. In the context of EFG crystal growth having a die length of greater than 15 cm (corresponding to crystal width) and die width of at least 0.5 cm (corresponding to crystal thickness), it has been discovered that it is particularly important to have the center of the die at an elevated temperature relative to the ends of the die, and have a temperature gradient that is uniform from the centerpoint to each of the ends. Not only are the relative temperatures of the middle and the ends of the die important, but also the gradients from the center of the die to the ends. Further, we have found that it is notably important to have a gradient that is not excessive, such as below 0.6°C/cm (see, e.g., claim 18). We discovered that in the migration from conventional crystal growing technology to larger-sized sheets, the conventional round crucibles inhibited our ability to achieve a uniform temperature gradient. In fact, it proved difficult to achieve the center of the die at a suitable processing temperature, let alone an appropriate temperature gradient to support successful EFG crystal growth having increased thickness and/or thickness uniformity.

7. Gradient Trim System. Further, as noted above, we have incorporated a particular gradient trim system. The incorporation and manipulation of the gradient trim system is notably important to achieve uniform thermal gradients, that is, to achieve a thermal gradient extending from the middle of the die to the left end of the die that is equivalent to the thermal gradient from the middle of the die to the right end of the die. During operation, as disclosed in the present specification, the thermal gradient trim system is adjusted based upon any non-uniformities between the two thermal gradients. Uniform thermal gradients are of particular importance in the successful growth of a large-sized, uniform thickness crystal that has uniform construction as quantified by a max  $\Delta_T$ . In particular, a non-uniform thermal gradient results in non-uniform growth of the crystal on the left side (center to left edge) relative to the right side (center to right side). This is particularly illustrated in crystal 80 shown in FIG. 6 of the present application, illustrating a notably large  $\Delta_T$  associated with non-uniform thermal gradients. Not only is a large  $\Delta_T$  generally undesirable but the large  $\Delta_T$  causes uncontrollable runaway process conditions

during processing. More particularly, it has been found that the growing crystal functions as a heat sink, notably directing heat from the more massive portions of the crystal into the environment and into the crystal growing apparatus. In the particular example shown in FIG. 6, the thicker, higher mass left portion of the crystal functions to remove more heat from the system relative to the thinner, slower growing side of the crystal (right side). This causes a runaway process condition in which the crystal that is grown is very non-uniform, the left side becoming thicker and thicker, the left side being notably thin. Indeed, the process conditions may reach a point at which the crystal that is grown is completely unusable and of low thickness. Accordingly, the gradient trim system plays a notable role in the achievement of large-sized single crystal sheets having the width and thickness dimensions of claims 1 and 11, the low thickness variation of claims 6 and 11, and the uniform construction quantified by  $\Delta_T$  of claim 12.

8. Heat Shield Assembly. Further, addressing the particular heat shield assembly to provide a baseline thermal profile, my co-inventors and I developed the disclosed heat shield system to provide a desirable baseline thermal profile to enable the manufacture of the claimed crystals. Particularly, the prior art shield assemblies are typically formed of a series of co-planar sheets, each having the *same* length and width. In contrast, as shown in FIG. 5, the shield assembly 26 has a stepped configuration. My co-inventors and I have discovered that in the context of growing high quality, next-generation single crystal sapphire sheets, it became quite important to achieve a baseline profile that minimizes thermal profile adjustments during processing. Indeed, use of the prior art shield assembly having a non-stepped configuration resulted in thermal profiles which prevented us from successfully growing large-sized sapphire single crystals having notable thickness (claims 1 and 11), low thickness variation (claims 6 and 11) and controlled  $\Delta_T$  (claim 12).

9. The prior art. The disclosure of Takahashi et al. is no more relevant than the state of the art over which the claimed invention has been developed. The most pertinent disclosure of the reference appears to be provided in connection with Example 3 describing a sapphire monocrystal block having the dimensions of 10 cm X 10 cm X 3 cm. Certainly, the single crystal block of Takahashi et al. does not meet the geometric features of the claimed invention. In this respect, the PTO has argued that it would have been obvious to one of ordinary skill in the art to

modify the dimensions of Takahashi et al. to meet those presently claimed. However, Takahashi et al. do not even remotely *enable* such a modification. As described above, multiple process and structural innovations have been developed by us through extensive research and development to enable formation of large-sized and uniform single crystal sapphire sheets. Clearly, there is no teaching of high aspect ratio crucibles, use of any particular stationary heat shield structure to provide a baseline thermal profile, utilization of a gradient trim system, or any alternative technology to achieve the claimed invention.

10. Summary. Among the features disclosed in the present application, the above-mentioned features drawn to use of a high aspect ratio crucible, use of a gradient trim system, and use of a particular heat shield assembly, each contribute to a successful formation of large-sized single crystal sheets having the claimed thickness and structural uniformity of the present claims. In fact, it was found that mere scaling of prior art technology which relies on round crucibles, limited or no adjustability to the thermal gradients, and/or non-stepped thermal shield assemblies, was not successful in the formation of the desirably large-sized uniform sheets of the claimed invention. The foregoing innovations were created by my co-inventors and I through extensive research and development, and are at least partly the result of empirical studies on the notable engineering hurdles associated with the successful growth of the claimed sapphire single crystals.

7. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further, that these statements were made with the knowledge that willful false statements and the like, so made, are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted,

7-10-2006  
Date

John W. Locher

